

Petrologic relationship between lamprophyres, carbonatites, and heavy rare-earth element enriched breccias at Hicks Dome

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Abstract

New petrological, geochemical, and *P–T* modelling results from igneous samples clarify how carbonatite-lamprophyre magmatism, fluorite and rare earth element (REE) enrichment are petrogenetically related in southern Illinois. *P–T* modelling reveals that igneous rocks derive from a deep mantle carbonated source, that is consistent with trace element signatures for a fluorine-rich transition zone origin. Major element systematics suggests liquid-immiscibility with lamprophyric melts as the origin for Ca-carbonatites. Heavy REE (HREE) enrichments in Hicks Dome breccias likely formed through preferential partitioning and transport of HREE by brine-melts, exsolved from a deep carbonatite body. Brine-melts redistributed HREEs throughout the system along brecciated pathways where they reprecipitated as HREE-rich phosphate/fluorocarbonate minerals (e.g. xenotime, florencite, synchesite) in host bedrock. The diversity of igneous rocks in southern Illinois highlights the area as an excellent natural laboratory to study carbonated melt petrogenesis and evolution.

1 | INTRODUCTION

Carbonatites and related ultramafic rocks represent some of the most puzzling rocks on Earth (Anenburg et al., 2021; Chakhmouradian, 2006; Yaxley et al., 2022). Carbonatites are mantle-derived rocks, though their petrogenesis, evolution, and association with REE and fluorite mineralization remains in debate (Kamenetsky et al., 2021). Carbonatites occur in different tectonic settings including cratons, continental rifts, orogenic belts, and oceanic islands. They also occur with other silica-undersaturated igneous rocks such as lamprophyres (Tappe et al., 2005). Most carbonatites are restricted to continental crust where thickened lithosphere is favourable to the production of CO₂-rich melts (Humphreys-Williams & Zahirovic, 2021). These melts originate from radiogenically enriched mantle domain endmembers such as enriched mantle

type 2 (EM2) and HIMU (high μ , where $\mu = {}^{238}\text{U}/{}^{204}\text{Pb}$). The HIMU reservoir, which is thought to reside in the mantle transition zone (MTZ); Huang et al. (2020), where anhydrous minerals (olivine) can also host appreciable levels of fluorine (F) in its crystal structure at high pressures (13–22 GPa). It is important to note that the postulated mantle source components (e.g. HIMU, EM) is only possible if interaction and chemical exchange with the overlying SCLM and continental crust can be excluded, but typically, carbonatite magmas rising through a thick lithosphere are always contaminated to some extent (Chandra et al., 2019).

Hicks Dome is a Permian alkaline-ultramafic intrusive complex in Hardin County, Illinois and is the centrepiece of the elusive Wauboukigou Igneous Province (WIP; Brown et al., 1954; Lewis & Mitchell, 1987; Reynolds et al., 1997; Denny et al., 2010; Denny & Seid, 2014; Shavers et al., 2016). These rocks occur as dikes,

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plugs and intrusive breccias (Bradbury & Baxter, 1992; Brown et al., 1954; Lewis & Mitchell, 1987) and are genetically linked to the fluorite mineralization in the Illinois-Kentucky Fluorspar district (Kendrick et al., 2002; Plumlee et al., 1995). Hicks Dome is the largest structure in the WIP. Intrusive rocks are hosted in middle-late Palaeozoic cratonic strata; emplacement of intrusive rocks is believed to be associated with volatile-rich magmatism (Bradbury & Baxter, 1992; Brown et al., 1954; Denny et al., 2015; Goldhaber et al., 1992).

Rocks at Hicks Dome and elsewhere in the WIP are alkaline, ultramafic, carbonate-rich, and show geochemical and petrological similarity to lamprophyres. However, the petrogenesis of these rocks with respect to temporally related fluorite and associated REE mineralization remains unclear (Lewis & Mitchell, 1987; Maria et al., 2019). Here, we present new petrologic and geochemical data from Hicks Dome drill cores and as well as samples and cores from several new locations in the region (Figure 1a) to clarify the origin of carbonatite emplacement and its relationship to Heavy REE (HREE) mineralization to elucidate their origin.

2 | BACKGROUND

Bradbury and Baxter (1992) classified several breccia pipes at Hicks Dome as carbonatitic, and Maria et al. (2019) classified the Grant Intrusive breccia pipe as a lamprophyre-carbonatite tuffsite. Sudden venting of diatremes lowered the pressure and temperature of the fluids possibly facilitated mineralization in breccias at Hicks Dome. A recent airborne magnetic survey (Figure 1a) reveals numerous magnetic anomalies in the WIP. Positive magnetic anomalies are surrounded by low negative magnetic anomalies (McCafferty & Brown, 2020) and are interpreted as igneous carbonatitic bodies (positive anomalies) whose exsolved fluids permeated and metasomatized (finitized) surrounding country rock (negative anomalies) and demagnetized its original signal, similar to the classic carbonatite occurrences in Scandinavia including the Alnö Carbonatite Complex (Kresten & Troll, 2018). These large and largely unexplored anomalies (Figure 1a) in southern Illinois and western Kentucky underscore the economic potential of the PWIP as a significant REE-bearing region as well as a natural laboratory to study carbonated melts.

The WIP in Illinois hosts igneous rocks including lamprophyre diatremes, sills, and dikes within the Reelfoot Rift and Rough Creek Grabens (Figure 1a), near Hicks Dome in Illinois and in western Kentucky (Bradbury & Baxter, 1992; Brock & Heyl Jr., 1961; Denny & Seid, 2014; Diller, 1885). These Illinois aulacogen-related structures formed during the Neoproterozoic-Cambrian rifting of Rodinia (Freiburg et al., 2020, 2022). Episodic reactivation of basement faults deformed overlying Palaeozoic strata and produced one of the most structurally complex areas in the Appalachian-Ouchita foreland (Craddock et al., 2018). Isotopic dating of igneous materials yields a Permian age of ~272 Ma (Reynolds et al., 1997).

Hicks Dome is centered within the Illinois-Kentucky Fluorspar District. The source of F and REE in the district is attributed to

Significance statement

Rare earth element enrichment at Hicks Dome and regional fluorite emplacement are petrogenetically related to carbonatite-lamprophyre magmatism and evolution originating from the deep mantle, possibly the transition zone.

the input of magmatic-derived fluid from deeply buried carbonatite bodies into the regional Mississippi Valley-Type (MVT) system (Morehead, 2013). Recent fluid inclusion studies indicate that ore fluids were acidic with a pH of 0–1.4, much lower than the typical 4–5.5 pH range of MVT fluids (Kenderes & Appold, 2017). Homogenization temperatures of coeval sphalerite fluid inclusions range from ~60 to 150°C and are consistent with late-stage crystallization from saline-rich magmatic-derived fluids.

3 | METHODOLOGY

Major and trace element analyses were performed by the US Geological Survey in Denver, CO using the lithium-borate fusion technique for inductively coupled plasma mass spectrometry (ICP-MS). Geochemical data and standard statistics are included in the supplementary files.

Petrologic modelling to determine P - T melt equilibration conditions was performed following the approach of Sun and Dasgupta (2020). This method describes a new thermobarometer for silica-poor, CO_2 -rich melts calibrated from high-temperature, high-pressure experimental melting data. Equations (1) and (2) from Sun and Dasgupta (2020) for equilibration pressure and temperature are:

$$P = 13 - \sqrt{\frac{T}{45.90} \left[\ln \left(\frac{\text{Al}_2\text{O}_3}{\text{MgO}} \right) - \Theta \right] - \frac{\Phi}{45.90} + 169} \quad (1)$$

$$T = \frac{10^4}{\Omega - 0.34\sqrt{P} - 1.26\ln(\text{MgO})} \quad (2)$$

The barometer is calibrated based on a new observation of pressure-dependent variation of Al_2O_3 in partial melts in equilibrium with garnet and olivine. Al_2O_3 contents of experimental melts derived from garnet-peridotites decrease systematically with the increase of P . This provides a basis for barometer calibration distinct from traditional silica-activity based barometers. As the major alumina-bearing phase in deep mantle sources, garnet presumably buffers Al_2O_3 in the melt through exchange reactions with other phases. This assumption is valid for natural silica-poor melts as they are thought to derive via partial melting of garnet-peridotite sources (Dasgupta et al., 2007; Sun & Dasgupta, 2020).

The Sun and Dasgupta (2020) thermometer is calibrated based on olivine-melt-Mg-exchange that governs the variations of MgO abundances in the melt as a function of T (Roeder & Emslie, 1970).

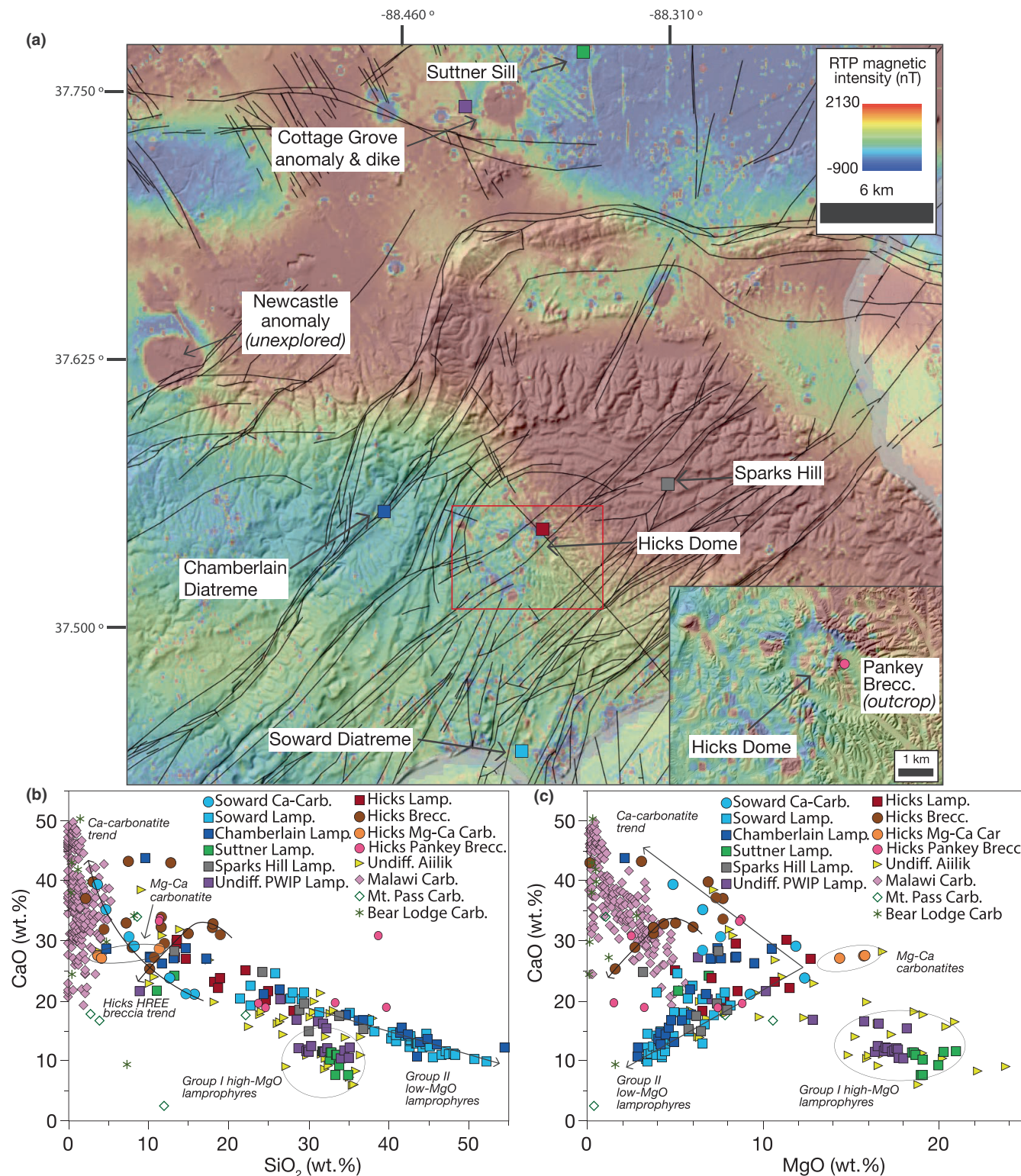


FIGURE 1 (a) Aeromagnetic map of the PWIP showing sample locations and prominent anomalies (aeromagnetic data collected by USGS; McCafferty & Brown, 2020). (b) Plot of CaO versus SiO₂. (c) Plot of CaO versus MgO. Additional data from Broom-Fendley et al. (2017, 2021); Denny et al. (2015), Maria et al. (2019), Tappe et al. (2006) and Verplanck et al. (2016).

Primary melt compositions are estimated by adding or subtracting olivine incrementally until the magma achieves chemical equilibrium with a mantle composition (Lee et al., 2009; Putirka et al., 2011).

Determining volatile (e.g., CO₂ and H₂O) concentrations in primary CO₂-rich melts continues to be challenging. As shown in several experimental studies on CO₂-bearing peridotite systems (Dasgupta

et al., 2007, 2013; Sun & Dasgupta, 2020), CO₂ abundances in the melts manifest an inverse correlation with SiO₂ concentration. The following expression empirically predicts CO₂ concentration in the garnet-peridotite partial melts with ≤ 48.6 wt% SiO₂: $\text{CO}_2 = 43.77 - 0.90 \times \text{SiO}_2$. CO₂ and SiO₂ are both in wt% on a H₂O-free basis.

4 | RESULTS

Major and trace element concentrations in WIP rocks show five geochemical groupings, a strong correlation in CaO and SiO₂ and distinct groupings in CaO-MgO space (Figure 1b,c). Lamprophyres and carbonatites broadly resemble the range of compositions of the Aillik Bay lamprophyre/carbonatite suite, which we consider to be analogs to the WIP suite (Maria et al., 2019; Tappe et al., 2006). Several samples from the Soward Diatreme and Hicks Dome contain high-CaO (>20%) and low SiO₂ (<12%) and are thus carbonatites.

Lamprophyres plot in two groups (Group I and II), contain an abundance of volatile-rich phases including melilite, phlogopite, amphibole, and calcite, and are dominated by Ca-Al rich phases and variable amounts of Nb-bearing rutile. Accessory fluorite occurs in both groups as do xenocrysts of olivine, pyroxene and garnet. Group I lamprophyres are ultramafic, while Group II lamprophyres show low-MgO (<12%) and a positive correlation between CaO and MgO (Figure 1c). The Mg-Ca carbonatite (from drill hole F2, Hicks Dome) is dominated by coarse, euhedral ankerite (>90%) and contains fluorite associated with a Th and LREE-rich interstitial phase (possibly Ce-britholite). The Ca-carbonatite (from Soward Diatreme) has high CaO (>20%), low MgO (<12%), and low SiO₂ (<15%). Soward carbonatite samples show a negative CaO and MgO trend and intersect the Group II lamprophyre trend at ~12% MgO and ~25% CaO. Hicks Dome HREE-enriched breccias are a chaotic and heterogeneous mix of brecciated country rock, fluorite, and various REE-bearing phosphates and fluorcarbonates.

Petrologic modelling reveals that Hicks Dome, Soward Diatreme, and the Sparks Hill lamprophyres (Group II) and carbonatites equilibrated at pressures and temperatures between 2 and 4 GPa and 1000 and 1150°C, respectively (Figure 2a). Group I ultramafic WIP melts equilibrated between pressures and temperatures between 4 and 5 GPa and 1400–1600°C, respectively (Figure 2a). Using parameterized experimental carbonated-peridotite data, we estimate that rocks in the district contained as much as 40 wt.% CO₂.

REE systematics and chondrite-normalized REE patterns further exemplify the compositional heterogeneity of these rocks (Figures 2 and 3). Lamprophyres and carbonatites show enrichments in REE, specifically LREEs (Figures 2 and 3). Group I lamprophyres are more depleted in HREE than Group II (Figure 2b). Both Ca and Mg-Ca carbonatites have higher total LREE with respect to lamprophyres and HREE-breccias (Figure 3a). HREE-breccias typically show low total abundances of LREE (Figure 3b), similar to other REE-breccia prospects around the world including Lofdal in Namibia, Bear Lodge in Wyoming, and Songwe Hill in Malawi (Broom-Fendley et al., 2017,

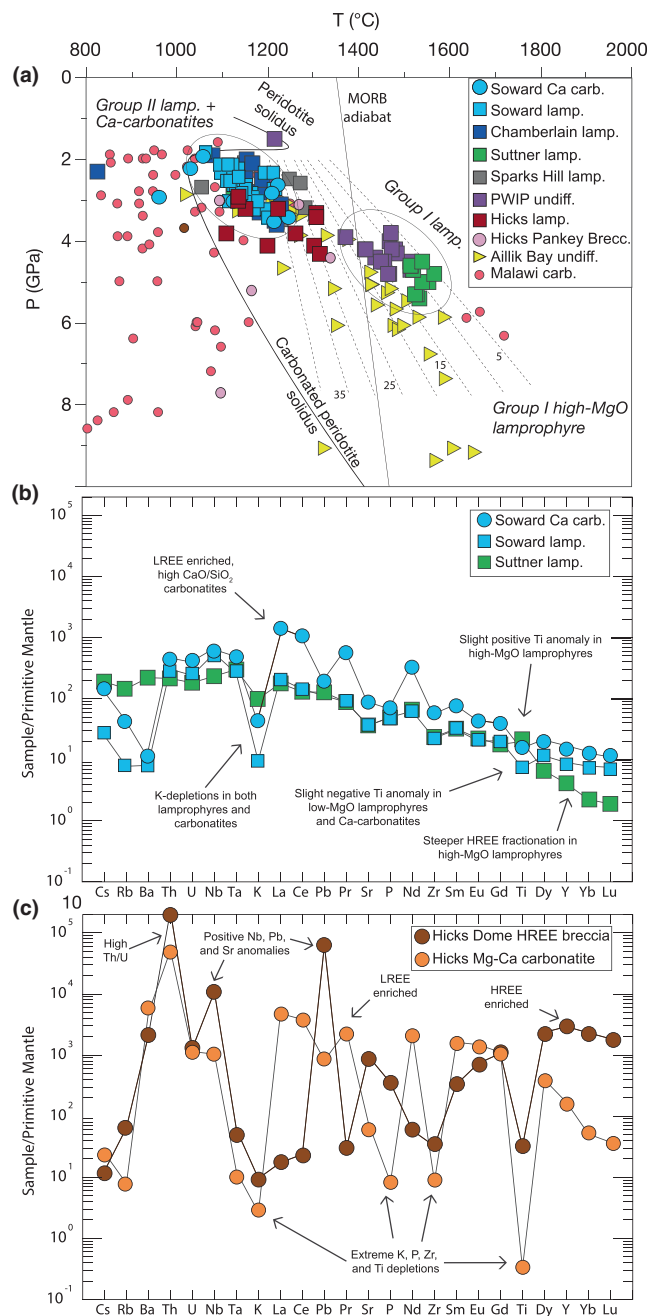


FIGURE 2 (a) *P*-*T* petrologic modelling results showing melt equilibration conditions for the PWIP. Model results and dashed CO₂ isopleths from Sun and Dasgupta (2020). (b) Spider diagram of Soward Ca-carbonatite (SO-8S-1653.5), Group II lamprophyre (SO-8S-1789.6) and Group I lamprophyre from Suttner Sill (S4B-183). (c) Spider diagram of Hicks Dome Mg-Ca carbonatite (F2-2391) and HREE-enriched breccia (FSB-38-2514). Additional data from Broom-Fendley et al. (2017, 2021); Denny et al. (2015); Maria et al. (2019); Tappe et al. (2006); Verplanck et al. (2016). Trace elements normalized using (Sun and McDonough) values. Solidus for volatile-free, fertile peridotite (Hirschmann, 2000) and carbonated peridotite from Dasgupta et al. (2007).

2021; Simonnetti & Bell, 1994). HREE-breccias are also unique in that they contain high Sc and Y (up to 600 ppm and 13,000 ppm, respectively).

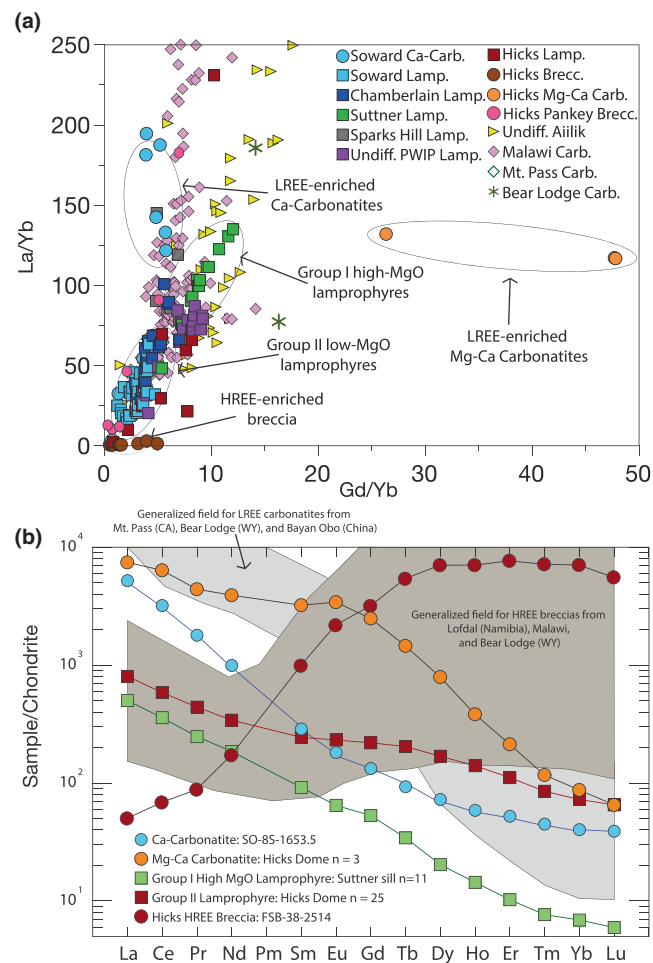


FIGURE 3 (a) Diagram of La/Yb vs. Gd/Yb highlighting the five distinct geochemical groupings in the PWIP. (b) Chondrite-normalized (Sun and McDonough, 1995). REE diagram. Note the similarity between LREE-enriched carbonatites and global occurrences. Likewise, Hicks Dome HREE breccia show strong similarity to other global REE-bearing breccia occurrences. Additional data from Broom-Fendley et al. (2017, 2021) Denny et al. (2015); Maria et al. (2019); Tappe et al. (2006); Verplanck et al. (2016).

5 | DISCUSSION

5.1 | A deep source for REE-enriched carbonatites

P-T modelling (Figure 2a), and the highly depleted HREE patterns of the lamprophyres and carbonatites (Figure 3) indicates a carbonated-mantle origin within the garnet stability field as the WIP magmatic reservoir. Group II lamprophyres show lower MgO than Group I lamprophyres and a Mg disparity exist between these two groups (Figure 1c). Group II lamprophyres correspond with Group I lamprophyres that lost MgO and CaO to melt-wall rock reactions during ascent (Tappe et al., 2006). The ultramafic composition and steep negative slope in HREE (Figure 3b) indicate that Group I lamprophyres represent the most primitive silicate mantle-derived melts in the WIP.

Ca-carbonatites and Group II lamprophyres in the WIP are likely conjugate pairs formed by liquid-immiscibility driven by crystal fractionation and degassing during ascent (Figure 4a). Ca-carbonatite/Group II lamprophyres major element compositions are consistent with other global occurrences that formed through liquid-immiscibility (Berndt & Klemme, 2022). Group II lamprophyres further evolved through fractional crystallization and crustal contamination. *P-T* modelling shows an overlap between both Group I and II varieties, indicating immiscibility between 1 and 2 GPa (Figure 2a). The Ca-carbonatites are like Group II lamprophyres with respect to trace elements (Figure 2b), though they show markedly higher concentrations in LREE (Figure 3a). This reflects preferential partitioning of LREE into the carbonatites. The Ca-carbonatites show depletions in Zr, K and Ti, though not to the degree as the Mg-Ca variety at Hicks Dome (Figure 2c).

The Mg-Ca carbonatites represent small amounts of primary mantle-derived carbonatite melt that interacted with shallow crustal carbonate rocks. Depletions in K and Ti may indicate a melt origin in the presence of MTZ phases such as liebermannite and perovskite (Roberge et al., 2015), which effectively sequester K and Ti relative to dry mantle melts.

At Hicks Dome, the majority of HREE are sequestered in xenotime and other HREE-bearing phases including synchysite, florencite, cheralite, britholite, pyrochlore, bastnäsite, and monazite (e.g. Denny et al., 2015, 2017). Trace element patterns diatreme breccias show elevated HREE concentrations with respect to LREE like REE-bearing breccias in Malawi, Namibia, and Wyoming, suggesting that common processes may concentrate HREE (Figure 4b) in such rocks. For example, HREE-enrichment in some peripheral zones at Bear Lodge in Wyoming is attributed to (1) fractionation during early crystallization of LREE selective minerals and Ca-REE fluorocarbonates, (2) REE liberation during breakdown of early crystallizing apatite and calcite with higher HREE/LREE ratios and (3) differential transport of REE in fluids with higher $\text{PO}_3^{4-}/\text{CO}_2^{3-}$ and F/CO_2^{3-} ratios (Andersen et al., 2017).

Experimental geochemistry has further demonstrated that LREEs partition to carbonatite melts that are either directly mantle-derived or immiscible from silicate melts. As carbonatite melts evolve, alkalis and LREEs concentrate in the residual melt due to their incompatibility in early crystallizing minerals. In many carbonatites, additional fractionation of calcite or ankerite leads to the evolution of the residual liquid into a mobile alkaline 'brine-melt' from which primary alkali REE carbonates form. These primary carbonates are rarely preserved owing to dissolution by later fluids and are replaced in-situ by monazite and alkali-free REE-fluorocarbonates (Anenburg et al., 2020). These experiments reveal that REEs were soluble only when alkalis were present. For example, Dy is more soluble than La when complexed with alkalis. These experiments reveal that complexing with alkalis is required for substantial REE transport in and around carbonatites as a precursor for economic grade-mineralization (Anenburg et al., 2020) and is likely responsible for elevated HREE concentrations in Hicks Dome near-surface breccias.

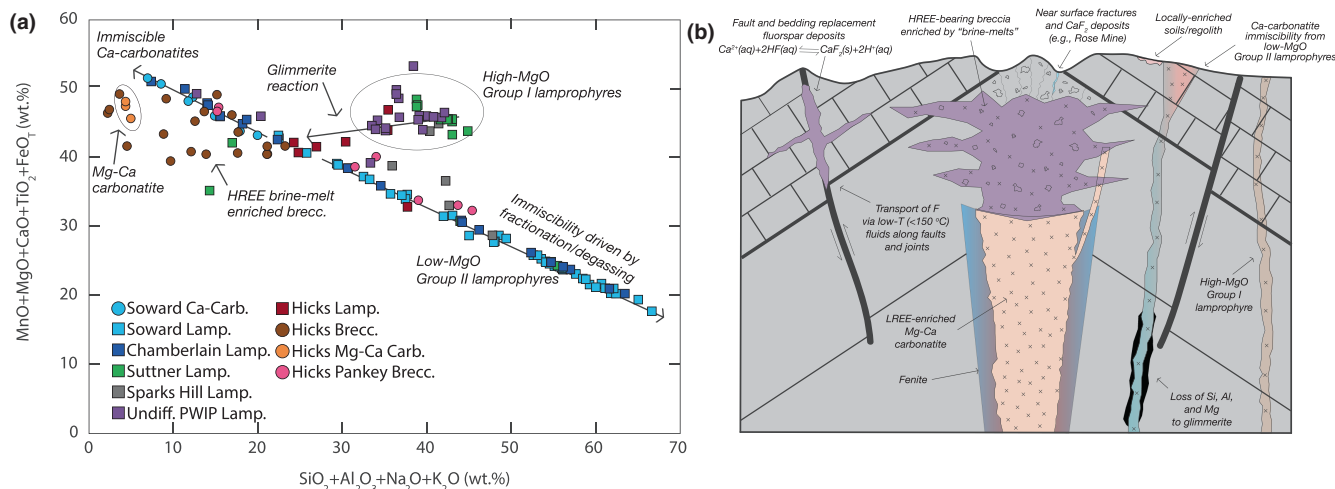


FIGURE 4 (a) Plot of elements compatible in carbonatite vs. carbonated-silicate melts showing that Ca-carbonatites in the PWIP likely derived through liquid immiscibility. Based on results from Berndt and Klemme (2022). Additional data from Broom-Fendley et al. (2017, 2021; Denny et al., 2015; Maria et al. (2019); Tappe et al. (2006); Verplanck et al. (2016). (b) Sub-surface schematic for the origin of PWIP melts, HREE-breccia and fluor spar deposits near Hicks Dome.

5.2 | Implications for REE deposits

Global REE deposits suggest a common origin for carbonatites from enriched mantle domains (enriched mantle type 1, EM1, EM2, and mantle domains labelled HIMU [high μ] type, where $\mu = {}^{238}\text{U}/{}^{204}\text{Pb}$). Based on new petrologic and geochemical results, WIP melts originated from a carbonated-mantle source and likely sampled at least two mantle reservoirs, and EM1 (Maria et al., 2019). Carbonate melts enriched in REEs originate from mantle sources that were previously re-fertilized by different components. For instance, it has been suggested that North American carbonatites, such as Bear Lodge in Wyoming, derive from an asthenospheric mantle plume that was composed of depleted mantle re-fertilized by a subduction-derived EM1 component Moore et al. (2015).

Radiogenic Sr and Nd work performed by Maria et al. (2019) suggests that most lamprophyres in the WIP show a HIMU signature like the Cook-Austral and Canary Islands, which host carbonatite derived melts (Hoernle et al., 2002). The HIMU-like source may reflect deeper contributions from recycled oceanic crust attributed to producing silica-undersaturated/carbonated melts observed in Bermuda (Mazza et al., 2019) and Cape Verde (Hoernle et al., 2002). The elevated F, Cl and S of HIMU sources act as fluxes and promote melting and transfer of REEs into carbonatite or carbonated silicate melt(s), which coalesce and rise to the crust during extension. Shallow immiscibility promotes the transfer of REEs from a carbonated silicate melt into carbonatite melt.

The HIMU reservoir (thought to reside in the MTZ) is the likeliest source for fluorine in the district. Previous studies (Roberge et al., 2015; Urann et al., 2017) show that nominally anhydrous minerals like olivine can host F in its crystal structure at MTZ pressures (13–22 GPa), which is partitioned into melt. HREE-enriched breccias show enrichment is related to late-stage dissolution, transport, and

precipitation of HREE from post-magmatic carbonatite-derived fluids (Anenburg et al., 2020).

6 | CONCLUSIONS

Carbonatites, lamprophyre, fluorite, and rare earth element hosted breccias all occur in southern Illinois and are petrogenetically related to one another. Group I lamprophyre represent the most primitive magma compositions in the district. Group II lamprophyres represent the Group I variety that has undergone melt wall-rock reaction (glimmeritization) during ascent through the crust. Our petrologic modelling indicate that Ca-carbonatites are the immiscible conjugate pair to Group II, differentiated lamprophyre. The Ca-carbonatite and Group II lamprophyre became immiscible at ~ 1.5 GPa in the crust. HREE enriched breccias at Hicks Dome likely formed through preferential partitioning and transport of HREE by brine-melts, exsolved from a deep carbonatite body and facilitated through complexation with Na and K. Brine-melts redistributed HREEs throughout the brecciated overburden and reprecipitated as HREE-rich phosphate/fluorocarbonate minerals. The diversity of igneous rocks in southern Illinois highlights the potential of the region as a significant REE-bearing region and natural regional laboratory to study carbonated melt petrogenesis and evolution.

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DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the supplementary material of this article.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

Table S1. Major and trace element geochemistry of PWIP samples.

Table S2. Petrologic modelling results following the method of Sun and Dasgupta (2020).

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